

Analysis of Aromatic Hydrocarbons in Gasoline and Naphtha with the Agilent 6820 Series Gas Chromatograph and a Single Polar Capillary Column

Application

Petrochemical

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Abstract

The aromatic hydrocarbons in finished gasoline, gasoline blending feedstock, and straight-run naphtha were quantitatively analyzed using an Agilent 6820 gas chromatograph equipped with a flame ionization detector and a single polar capillary column. An Internal standardization method, using n-tridecane as the internal standard, yielded quantitative concentration data for 57 aromatic target compounds, with a relative standard deviation of less than 1% for almost all compounds.

Introduction

Motorization in China is growing rapidly, resulting in an increasing demand for gasoline. Since carcinogenic components exist within gasoline, there is a need to monitor and control its composition. Many regulatory standards were created worldwide for this purpose.

The People's Republic of China National Standards GB 17930-1999 [1] describes the quality of unleaded finished gasoline for motor vehicles. Maximum allowable concentrations for benzene and total aromatic hydrocarbon are 2.5 and 40 weight percent (wt %) respectively. This latter method uses a gas chromatograph (GC) equipped with a valve system and two packed columns (OV-101 and TCEP) to analyze benzene and toluene in gasoline.

In Europe and Japan, the benzene content of gasoline must be 1 wt % or less, and the total aromatic hydrocarbon content is limited by regulation.

There are approved methods for the analysis of aromatic hydrocarbons in gasoline from the American Society for Testing and Materials (ASTM). ASTM D5769 [2] is the standard test method for determining benzene, toluene, and total aromatics in finished gasoline by gas chromatography/mass spectrometry (GC/MS). This method requires a GC/MS system.

ASTM D5580 [3] is the standard gas chromatography (GC) test method for determining benzene, toluene, ethylbenzene, p/m-xylene, o-xylene, C9 and heavier aromatics, and total aromatics in finished gasoline. This method uses a valve system, two packed columns, and requires two separate analyses to completely measure the aromatic content in gasoline.



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ASTM D3606 [4] is the standard test method for determining benzene and toluene in finished motor and aviation gasoline by GC. Although this method can analyze for benzene and toluene, it is unable to analyze for C8 or heavier aromatic hydrocarbons.

Japanese Industrial Standards (JIS) K 2536-1996 [5] describes the use of a single TCEP or PEG-20M capillary column to analyze aromatic hydrocarbons in gasoline. This method requires no valve system or modification of the GC. While both columns are non-cross-linked liquid phase columns, they cannot withstand high-temperature conditions.

In this application note, the described method uses a single cross-linked polyethylene glycol capillary column for the GC analysis of aromatic hydrocarbons in finished gasoline and gasoline blending feedstock.

Experimental

In this work, an Agilent 6820 GC equipped with a split/splitless inlet and a flame ionization detector (FID) appear in Table 1.

Table 1. Analytical Conditions

GC	Agilent 6820 Gas Chromatograph
Inlet	Split/Splitless; 240 °C, split mode, split ratio 50:1
Carrier	Helium, 1.4 mL/min at 40 °C
Column	60 m, 0.25 mm id, 0.25 µm film HP-INNOWax (p/n 19091N-136)
Oven	40 °C (5 min); 5 °C /min; 240 °C (15 min)
Detector	FID, 240 °C; Makeup gas: Helium, 40 mL/min

Samples analyzed in this work include: Chinese finished gasoline (RON = 93), Japanese finished regular gasoline (RON = 90), Japanese finished

premium gasoline (RON = 100), reformat gasoline, heavy fluid catalytic cracking (FCC) gasoline, and straight-run heavy naphtha.

JIS K 2536-1996 describes a quantitative method for aromatic hydrocarbons using n-undecane as the internal standard. Since Chinese gasoline includes some n-undecane, a different internal standard (IS) was required. The alternate, n-tridecane, was used and about 2 wt % was added to each sample.

Each aromatic hydrocarbon component was identified using an Agilent 6890N/5973N GC/MS and analytical conditions were determined by method translation.

By consensus, theoretical response factors are used for the correction of the FID detector response of hydrocarbons (as determined by this method). The response of an FID to hydrocarbons is determined by the ratio of the molecular weight of the carbon in the analyte to the total molecular weight of analyte. The response factors, as listed in Table 2, are relative to those calculated for heptane. Calculations are based on the following equation:

$$F_h = \frac{(C_{aw} \times C_n) + (H_{aw} \times H_n)}{C_{aw} \times C_n} \times 0.83905$$

Where: Fh = Relative response factor for a hydrocarbon type group of a particular carbon number

C_{aw} = Atomic weight of carbon, 12.011

C_n = Number of carbon molecules in the group

H_{aw} = Atomic weight of hydrogen, 1.008

H_n = Number of hydrogen molecules in the group; 0.83905 is the correction factor with heptane as unity (1.0000). 0.7487 is used with methane as unity.

Table 2. Theoretical FID Relative Response Factors

Carbon number	Saturated paraffins	Olefins	Saturated naphthenes	Unsaturated naphthenes	Aromatics
1	1.1207	—	—	—	—
2	1.0503	—	—	—	—
3	1.0268	0.9799	—	—	—
4	1.0151	0.9799	—	—	—
5	1.0080	0.9799	0.9799	0.9517	—
6	1.0034	0.9799	0.9799	0.9564	0.9095
7	1.0000	0.9799	0.9799	0.9598	0.9195
8	0.9975	0.9799	0.9799	0.9623	0.9271
9	0.9955	0.9799	0.9799	0.9642	0.9329
10	0.9940	0.9799	0.9799	0.9658	0.9376
11	0.9927	0.9799	0.9799	0.9671	0.9415
12	0.9916	0.9799	0.9799	0.9681	0.9447
13	0.9907	0.9799	0.9799	0.9690	0.9474
14	0.9899	0.9799	0.9799	0.9698	0.9497
15	0.9893	0.9799	0.9799	0.9705	0.9517

Results and Discussion

A chromatogram of reformate gasoline is shown in Figure 1. Reformate is a high concentration, aromatic hydrocarbon gasoline feedstock from the reformation process. There are low boiling point nonaromatic hydrocarbons eluted before benzene. High boiling point nonaromatic hydrocarbons were not found in reformate, and all peaks after benzene were aromatic hydrocarbons. A peak of n-tridecane (peak number 19) elutes at 19.45 minutes, between 1,2,4-trimethylbenzene and 1,4-diethylbenzene. These peaks separate at the base line. The n-tridecane peak does not overlap with any aromatic hydrocarbon peak.

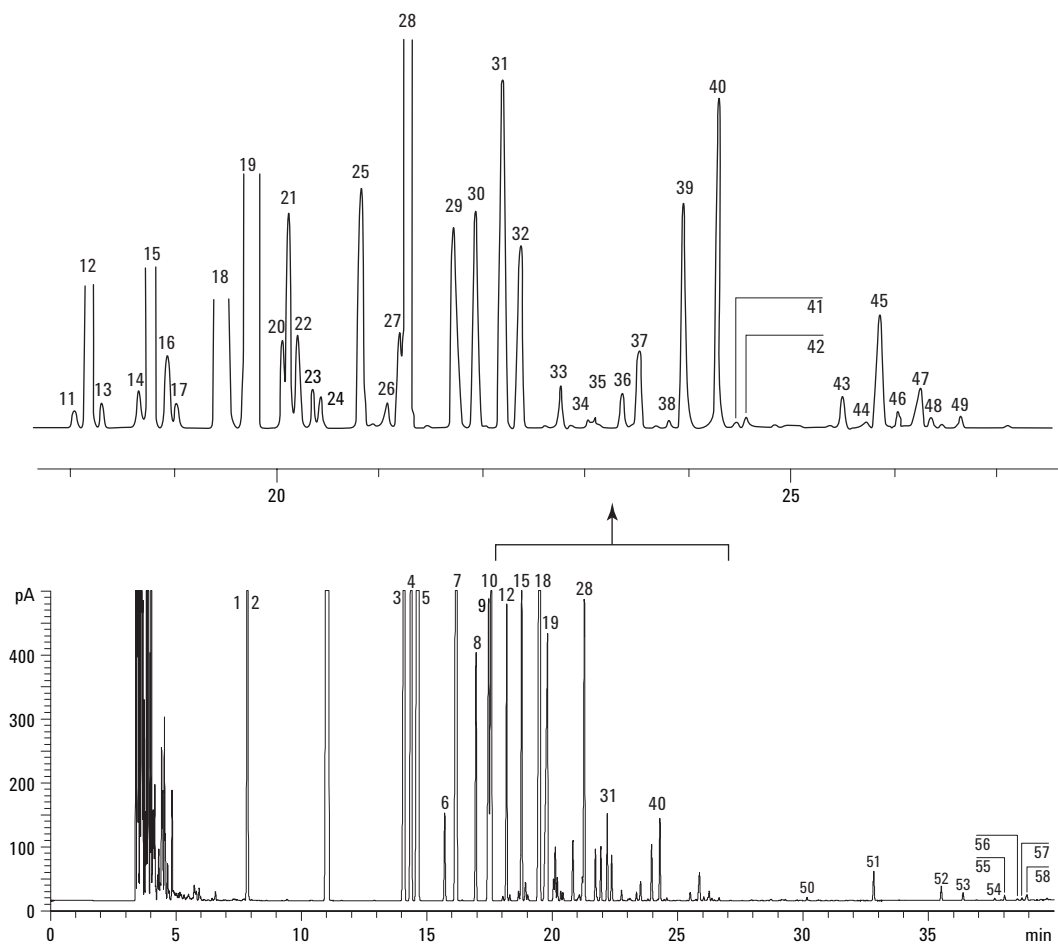


Figure 1. Chromatogram of reformate.

A chromatogram of heavy FCC gasoline is shown in Figure 2. FCC gasoline contains paraffin, olefin, naphthene, and aromatic hydrocarbons. There are some small interfering nonaromatic hydrocarbon peaks among benzene and ethylbenzene. It also seems that benzene and toluene overlap another nonaromatic hydrocarbon, but the peak area of this interference is relatively small.

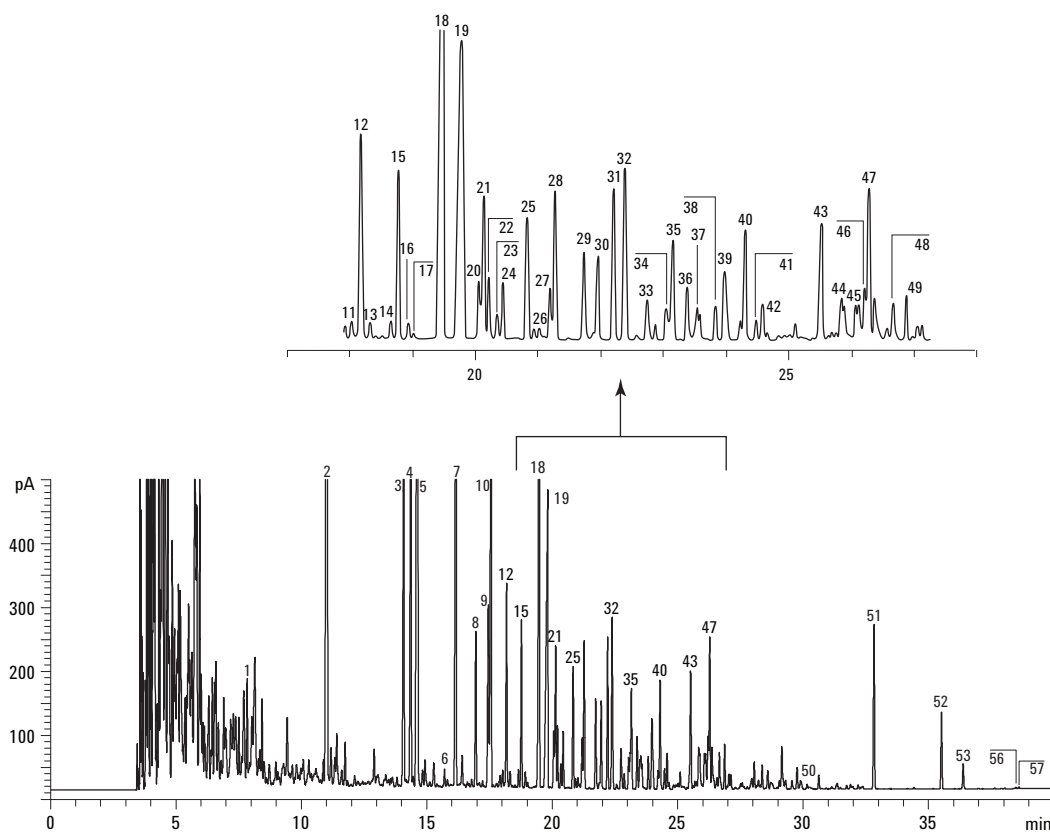


Figure 2. Chromatogram of heavy FCC gasoline.

A chromatogram of heavy naphtha is shown in Figure 3. Naphtha contains paraffin, naphthene, and aromatic hydrocarbons. There are some nonaromatic hydrocarbon peaks near benzene and few after toluene.

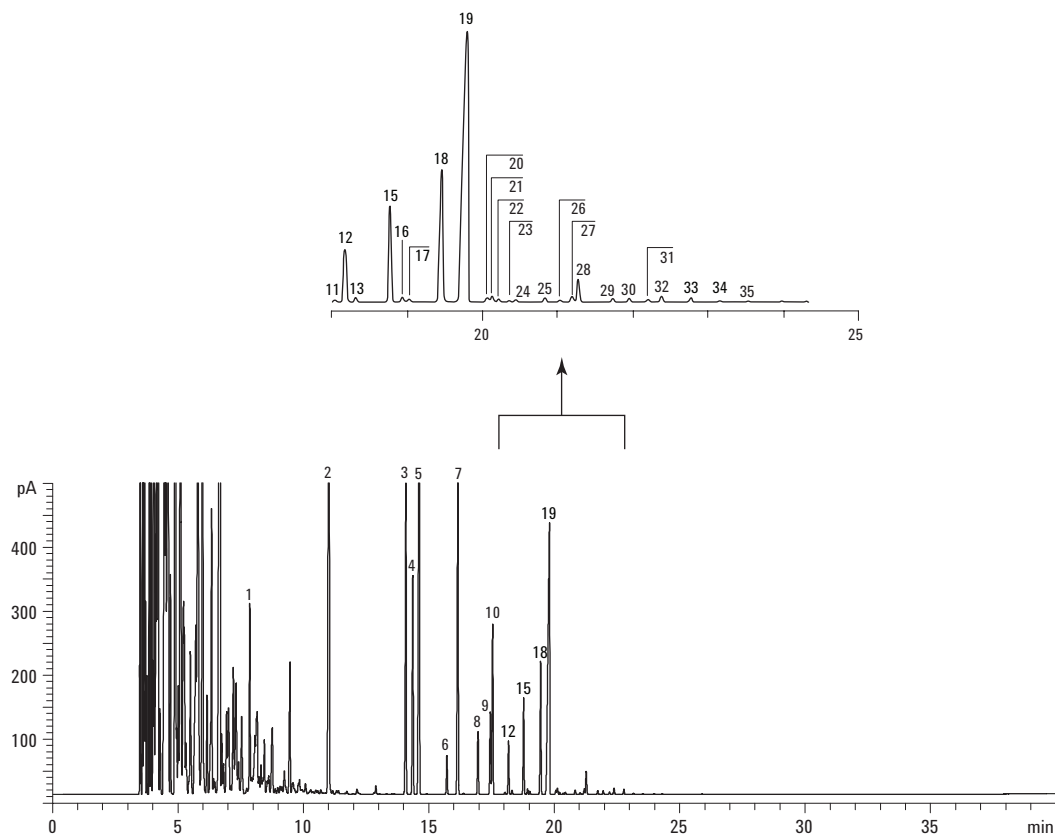


Figure 3. Chromatogram of heavy straight-run naphtha.

A chromatogram of Japanese finished premium gasoline (RON = 100) is shown in Figure 4. The premium gasoline is normally blended with reformate, light FCC gasoline, alkylate, isomerate, butane, butene, and some oxygenates. The premium gasoline does not contain a heavy fraction of nonaromatic hydrocarbons. Only a few small peaks of nonaromatic hydrocarbons are found near the benzene peak.

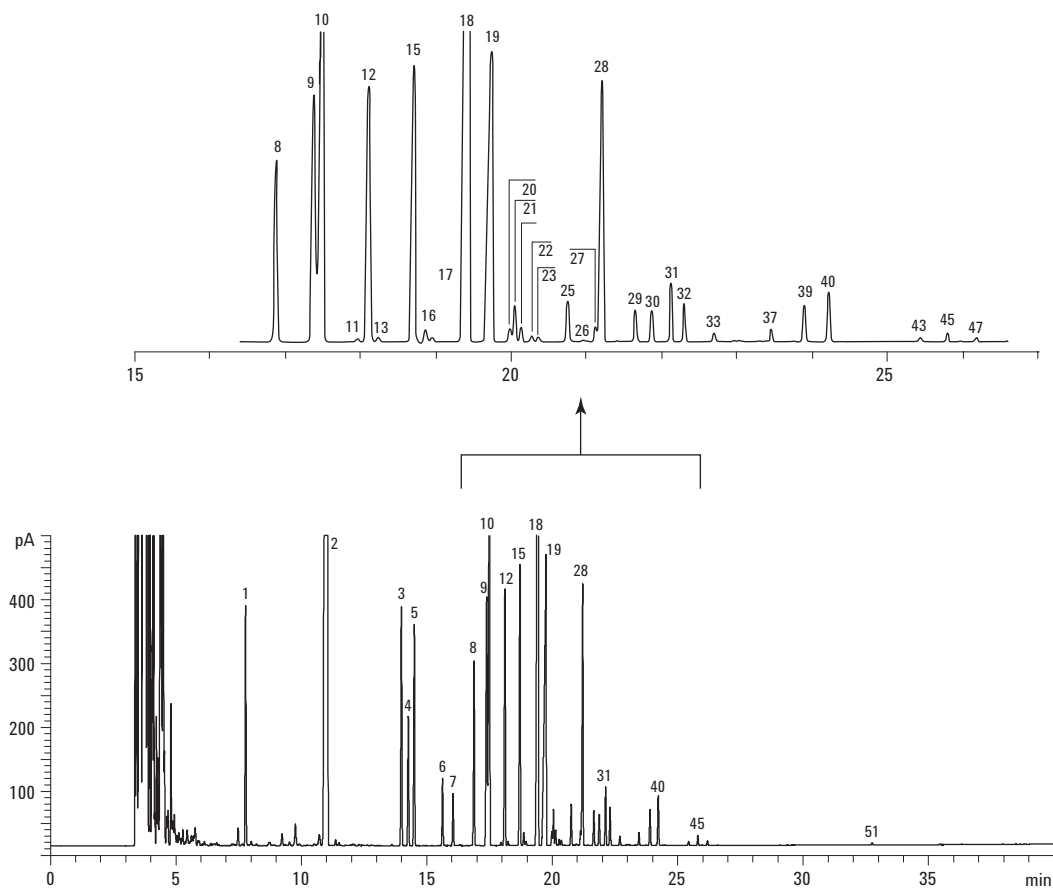


Figure 4. Chromatogram of Japanese finished premium gasoline.

A chromatogram of Japanese finished regular gasoline (RON = 90) is shown in Figure 5. The regular gasoline is normally blended with reformate, heavy FCC gasoline, light naphtha, butane, and butene. In this case, there are some heavy nonaromatic hydrocarbons. These peaks elute between benzene and o-xylene. There is no nonaromatic hydrocarbon peak after o-xylene.

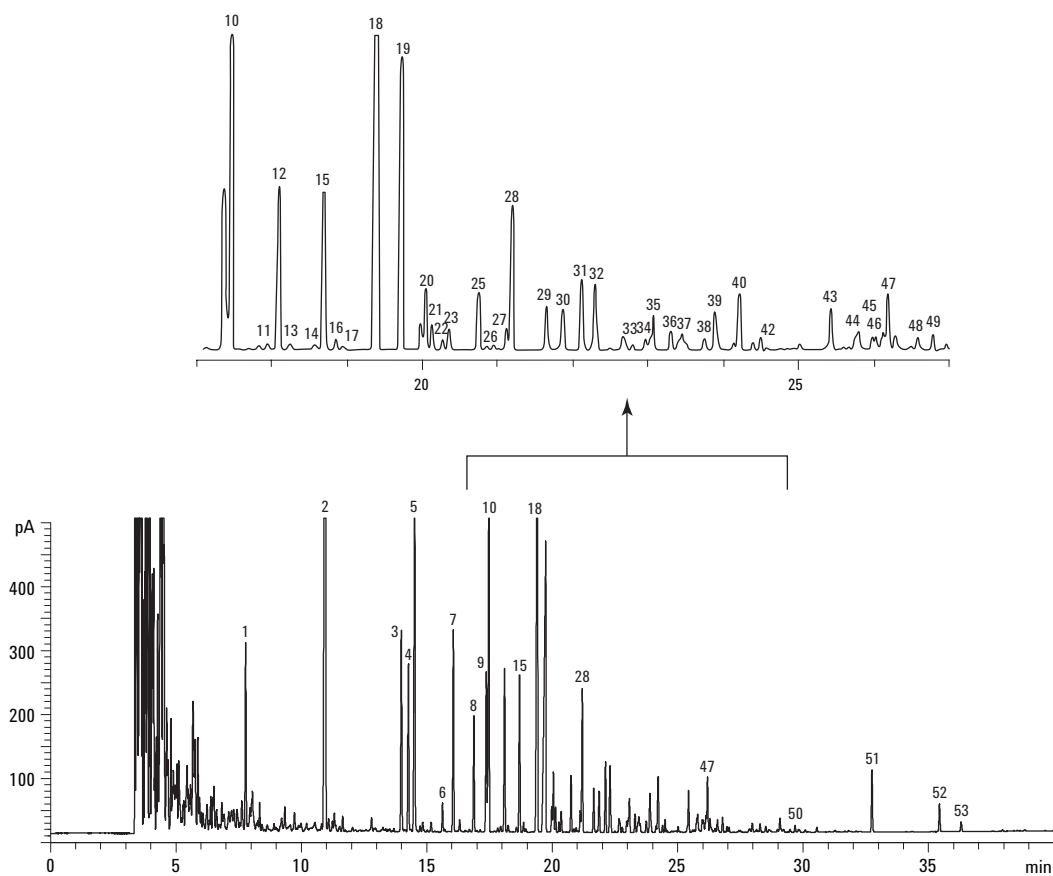


Figure 5. Chromatogram of Japanese finished regular gasoline.

A chromatogram of Chinese finished gasoline (RON = 93) is shown in Figure 6. This gasoline contains heavy fractions of nonaromatic hydrocarbons. The peak at 12.9 min is n-undecane. In this case, if n-undecane is used as an internal standard, the quantitative results will be incorrect. There is no nonaromatic hydrocarbon near n-tridecane.

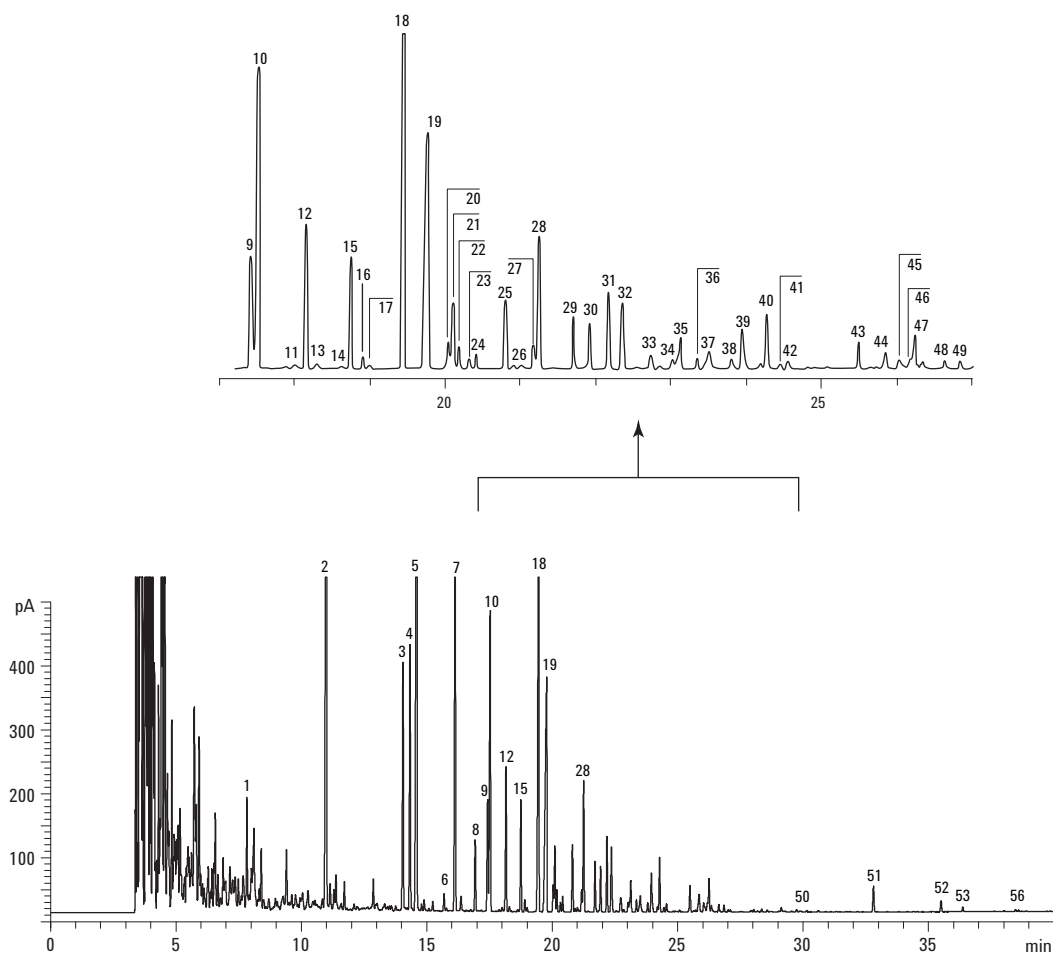


Figure 6. Chromatogram of Chinese finished gasoline.

Quantitative results for finished gasoline and gasoline feedstock are shown in Table 3. Reformate and heavy FCC gasoline contain heavy aromatic hydrocarbons such as biphenyl and dimethylnaphthalene. However, heavy straight-run naphtha does not contain any heavy fraction; tetramethylbenzenes and naphthalene are not detected.

Table 3. Quantitative Results for Finished Gasoline and Blending Feedstock

Peak number	Compound name	Reformate Wt %	Heavy FCC gasoline Wt %	Heavy naphtha Wt %	Japanese premium gasoline Wt %	Japanese regular gasoline Wt %	Chinese gasoline Wt %
1	Benzene	4.716	0.347	0.667	0.729	0.604	0.371
2	Toluene	15.705	6.141	3.466	22.345	5.744	3.550
3	Ethylbenzene	4.227	1.988	1.286	0.828	0.714	0.909
4	p-Xylene	3.794	1.797	0.878	0.439	0.582	0.951
5	m-Xylene	8.260	4.706	2.213	0.765	1.487	2.669
6	iso-Propylbenzene	0.332	0.068	0.152	0.229	0.101	0.061
7	o-Xylene	5.506	2.201	1.360	0.174	0.697	1.444
8	n-Propylbenzene	0.865	0.590	0.247	0.667	0.405	0.248
9	1-Methyl-4-ethylbenzene	1.350	0.819	0.332	1.094	0.630	0.411
10	1-Methyl-3-ethylbenzene	2.935	2.070	0.660	2.396	1.463	1.108
11	iso-Butylbenzene	0.018	0.070	0.009	0.013	0.025	0.019
12	1,3,5-Trimethylbenzene	1.069	0.812	0.210	0.938	0.577	0.521
13	sec-Butylbenzene	0.020	0.074	0.017	0.015	0.028	0.019
14	Styrene	0.036	0.073	0.000	0.000	0.020	0.009
15	1-Methyl-2-ethylbenzene	1.336	0.629	0.375	1.043	0.555	0.390
16	1-Methyl-4-iso-propylbenzene	0.071	0.061	0.022	0.049	0.036	0.044
17	1-Methyl-3-iso-propylbenzene	0.023	0.027	0.012	0.017	0.014	0.016
18	1,2,4-Trimethylbenzene	4.501	2.599	0.530	3.932	2.185	1.954
19	n-Tridecane (IS)	1.854	2.127	2.107	2.168	2.043	1.556
20	1,4-Diethylbenzene	0.073	0.241	0.018	0.122	0.211	0.099
21	1-Methyl-4-n-propylbenzene	0.179	0.534	0.025	0.050	0.081	0.232
22	1-Methyl-3-n-propylbenzene	0.073	0.201	0.009	0.020	0.034	0.072
23	1,3-Diethylbenzene	0.031	0.087	0.005	0.017	0.069	0.032
24	n-Butylbenzene	0.027	0.200	0.009	0.000	0.000	0.054
25	1,3-Dimethyl-5-ethylbenzene	0.212	0.471	0.016	0.148	0.012	0.246
26	alpha-Methylstyrene	0.031	0.013	0.007	0.006	0.005	0.006
27	1-Methyl-2-n-propylbenzene	0.069	0.196	0.022	0.044	0.077	0.079
28	1,2,3-Trimethylbenzene	1.120	0.566	0.095	0.963	0.516	0.484
29	1,4-Dimethyl-2-ethylbenzene	0.181	0.352	0.014	0.119	0.155	0.180
30	1,3-Dimethyl-4-ethylbenzene	0.188	0.373	0.013	0.105	0.156	0.167
31	1,2-Dimethyl-4-ethylbenzene	0.283	0.539	0.010	0.197	0.245	0.261

Table 3. Quantitative Results for Finished Gasoline and Blending Feedstock (continued)

Peak number	Compound name	Reformat Wt %	Heavy FCC gasoline Wt %	Heavy naphtha Wt %	Japanese premium gasoline Wt %	Japanese regular gasoline Wt %	Chinese gasoline Wt %
32	Indane	0.154	0.744	0.025	0.135	0.276	0.261
33	1,3-Dimethyl-2-ethylbenzene	0.035	0.190	0.020	0.032	0.018	0.077
34	Diethylmethylbenzene	0.006	0.120	0.000	0.000	0.040	0.034
35	1-Methyl-4- <i>iso</i> -butylbenzene	0.010	0.495	0.005	0.000	0.160	0.148
36	<i>iso</i> -Propenylbenzene	0.026	0.195	0.000	0.000	0.062	0.043
37	1,2-Dimethyl-3-ethylbenzene	0.062	0.122	0.000	0.043	0.024	0.087
38	1-Methyl-4- <i>iso</i> -butylbenzene	0.007	0.131	0.000	0.000	0.044	0.036
39	1,2,3,4-Tetramethylbenzene	0.192	0.382	0.000	0.126	0.174	0.170
40	1,2,3,5-Tetramethylbenzene	0.284	0.449	0.000	0.177	0.217	0.209
41	2,4-Diethyl-1-methylbenzene	0.004	0.081	0.000	0.000	0.044	0.016
42	1-Methyl-4- <i>tert</i> -butylbenzene	0.008	0.131	0.000	0.000	0.008	0.028
43	5-Methylindane	0.028	0.496	0.000	0.015	0.161	0.100
44	C11-Aromatic	0.004	0.030	0.000	0.000	0.009	0.005
45	1,2,4,5-Tetramethylbenzene	0.128	0.143	0.000	0.034	0.120	0.087
46	C11-Aromatic	0.013	0.137	0.000	0.000	0.048	0.051
47	4-Methylindane	0.039	0.610	0.000	0.016	0.206	0.123
48	C11-Aromatic	0.006	0.234	0.000	0.000	0.074	0.027
49	C11-Aromatic	0.009	0.173	0.000	0.000	0.057	0.028
50	Pentamethylbenzene	0.012	0.042	0.000	0.000	0.007	0.006
51	Naphthalene	0.105	0.664	0.000	0.008	0.233	0.097
52	2-Methylnaphthalene	0.051	0.309	0.000	0.000	0.106	0.043
53	1-Methylnaphthalene	0.029	0.104	0.000	0.000	0.038	0.019
54	2-Ethylnaphthalene	0.007	0.000	0.000	0.000	0.000	0.000
55	Dimethylnaphthalene	0.017	0.000	0.000	0.000	0.000	0.000
56	Biphenyl	0.005	0.006	0.000	0.000	0.000	0.007
57	Dimethylnaphthalene	0.009	0.008	0.000	0.000	0.000	0.000
58	Dimethylnaphthalene	0.029	0.000	0.000	0.000	0.000	0.000

Chinese finished gasoline and reformat were investigated for reproducibility of quantitative results. These data are shown in Tables 4 and 5, respectively. Most percent RSD are within 1%, except for a few peaks in very small concentration.

Table 4. Reproducibility Data for Chinese Finished Gasoline

Peak	Compound name	RT, min	Ave wt %	%RSD
1	Benzene	7.83	0.373	0.658
2	Toluene	10.99	3.567	0.658
3	Ethylbenzene	14.05	0.911	0.481
4	p-Xylene	14.33	0.953	0.438
5	m-Xylene	14.59	2.679	0.548
6	<i>iso</i> -Propylbenzene	15.68	0.061	0.259
7	o-Xylene	16.13	1.448	0.498
8	n-Propylbenzene	16.93	0.249	0.406
9	1-Methyl-4-ethylbenzene	17.42	0.412	0.526
10	1-Methyl-3-ethylbenzene	17.52	1.111	0.485
11	<i>iso</i> -Butylbenzene	18.00	0.019	0.285
12	1,3,5-Trimethylbenzene	18.15	0.523	0.454
13	<i>sec</i> -Butylbenzene	18.29	0.019	1.776
14	Styrene	18.62	0.009	0.754
15	1-Methyl-2-ethylbenzene	18.75	0.391	0.405
16	1-Methyl-4- <i>iso</i> -propylbenzene	18.91	0.044	0.333
17	1-Methyl-3- <i>iso</i> -propylbenzene	19.00	0.016	0.819
18	1,2,4-Trimethylbenzene	19.45	1.959	0.426
19	n-Tridecane (IS)	19.77	1.556	0.000
20	1,4-Diethylbenzene	20.03	0.099	0.308
21	1-Methyl-4-n-propylbenzene	20.10	0.232	0.393
22	1-Methyl-3-n-propylbenzene	20.19	0.072	0.377
23	1,3-Diethylbenzene	20.33	0.033	0.474
24	n-Butylbenzene	20.41	0.054	0.435
25	1,3-Dimethyl-5-ethylbenzene	20.81	0.246	0.369
26	<i>alpha</i> -Methylstyrene	21.00	0.017	0.315
27	1-Methyl-2-n-propylbenzene	21.17	0.079	0.367
28	1,2,3-Trimethylbenzene	21.25	0.485	0.345
29	1,4-Dimethyl-2-ethylbenzene	21.71	0.180	0.417
30	1,3-Dimethyl-4-ethylbenzene	21.93	0.168	0.342
31	1,2-Dimethyl-4-ethylbenzene	22.18	0.261	0.354
32	Indane	22.36	0.261	0.446
33	1,3-Dimethyl-2-ethylbenzene	22.72	0.077	0.412
34	Diethylmethylbenzene	23.02	0.034	0.282
35	1-Methyl-4- <i>iso</i> -butylbenzene	23.13	0.148	0.451
36	<i>iso</i> -Propenylbenzene	23.36	0.043	0.320
37	1,2-Dimethyl-3-ethylbenzene	23.51	0.087	0.496
38	1-Methyl-4- <i>iso</i> -butylbenzene	23.80	0.036	0.952
39	1,2,3,4-Tetramethylbenzene	23.95	0.170	0.245
40	1,2,3,5-Tetramethylbenzene	24.28	0.209	0.304
41	2,4-Diethyl-1-methylbenzene	24.45	0.016	0.268

Table 4. Reproducibility Data for Chinese Finished Gasoline (continued)

Peak	Compound name	RT, min	Ave wt %	%RSD
42	1-Methyl-4- <i>tert</i> -butylbenzene	24.55	0.028	0.527
43	5-Methylindane	25.49	0.100	0.356
44	C11-Aromatic	25.73	0.005	4.640
45	1,2,4,5-Tetramethylbenzene	25.86	0.088	0.602
46	C11-Aromatic	26.03	0.052	0.764
47	4-Methylindane	26.25	0.123	0.472
48	C11-Aromatic	26.34	0.028	1.321
49	C11-Aromatic	26.64	0.028	0.674
50	Pentamethylbenzene	30.14	0.006	1.083
51	Naphthalene	32.80	0.097	0.374
52	2-Methylnaphthalene	35.50	0.043	0.299
53	1-Methylnaphthalene	36.36	0.020	1.206
54	2-Ethylnaphthalene	37.64	—	—
55	Dimethylnaphthalene	38.03	—	—
56	Biphenyl	38.47	0.007	5.155
57	Dimethylnaphthalene	38.73	—	—
58	Dimethylnaphthalene	38.92	—	—

Table 5. Reproducibility Data for Reformate

Peak	Compound name	RT, min	Ave wt %	%RSD
1	Benzene	7.862	4.659	0.805
2	Toluene	11.086	15.556	0.581
3	Ethylbenzene	14.110	4.201	0.386
4	p-Xylene	14.395	3.772	0.369
5	m-Xylene	14.677	8.215	0.346
6	iso-Propylbenzene	15.716	0.331	0.375
7	o-Xylene	16.200	5.477	0.333
8	n-Propylbenzene	16.962	0.861	0.297
9	1-Methyl-4-ethylbenzene	17.465	1.347	0.235
10	1-Methyl-3-ethylbenzene	17.580	2.924	0.271
11	iso-Butylbenzene	18.032	0.018	0.771
12	1,3,5-Trimethylbenzene	18.188	1.065	0.283
13	sec-Butylbenzene	18.307	0.020	0.892
14	Styrene	18.661	0.035	1.926
15	1-Methyl-2-ethylbenzene	18.789	1.328	0.339
16	1-Methyl-4-iso-propylbenzene	18.933	0.070	0.555
17	1-Methyl-3-iso-propylbenzene	19.025	0.023	0.870
18	1,2,4-Trimethylbenzene	19.512	4.478	0.271
19	n-Tridecane (IS)	19.815	1.854	0.000
20	1,4-Diethylbenzene	20.052	0.073	0.490
21	1-Methyl-4-n-propylbenzene	20.120	0.179	0.662
22	1-Methyl-3-n-propylbenzene	20.203	0.073	1.586
23	1,3-Diethylbenzene	20.344	0.031	1.174
24	n-Butylbenzene	20.429	0.026	0.402
25	1,3-Dimethyl-5-ethylbenzene	20.823	0.212	0.276
26	alpha-Methylstyrene	21.076	0.031	0.853
27	1-Methyl-2-n-propylbenzene	21.196	0.069	0.548
28	1,2,3-Trimethylbenzene	21.282	1.114	0.322
29	1,4-Dimethyl-2-ethylbenzene	21.721	0.180	0.348
30	1,3-Dimethyl-4-ethylbenzene	21.939	0.187	0.281
31	1,2-Dimethyl-4-ethylbenzene	22.195	0.281	0.314
32	Indane	22.368	0.153	0.419
33	1,3-Dimethyl-2-ethylbenzene	22.763	0.035	1.199
34	Diethylmethylbenzene	23.032	0.006	4.721
35	1-Methyl-4-iso-butylbenzene	23.089	0.010	4.312
36	iso-Propenylbenzene	23.360	0.028	6.343
37	1,2-Dimethyl-3-ethylbenzene	23.520	0.064	2.826
38	1-Methyl-4-iso-butylbenzene	23.815	0.007	1.099
39	1,2,3,4-Tetramethylbenzene	23.962	0.192	0.325
40	1,2,3,5-Tetramethylbenzene	24.294	0.283	0.268
41	2,4-Diethyl-1-methylbenzene	24.463	0.004	0.785
42	1-Methyl-4-tert-butylbenzene	24.564	0.008	3.870
43	5-Methylindane	25.500	0.028	0.453
44	C11-Aromatic	25.732	0.004	3.307
45	1,2,4,5-Tetramethylbenzene	25.866	0.127	0.428
46	C11-Aromatic	26.042	0.013	1.102
47	4-Methylindane	26.255	0.039	0.295
48	C11-Aromatic	26.363	0.005	5.996

Table 5. Reproducibility Data for Reformate (continued)

Peak	Compound name	RT, min	Ave wt %	%RSD
49	C11-Aromatic	26.651	0.009	0.708
50	Pentamethylbenzene	30.158	0.012	0.550
51	Naphthalene	32.815	0.104	0.435
52	2-Methylnaphthalene	35.507	0.051	0.370
53	1-Methylnaphthalene	36.376	0.029	0.394
54	2-Ethylnaphthalene	37.636	0.007	0.544
55	Dimethylnaphthalene	38.033	0.017	0.957
56	Biphenyl	38.549	0.005	1.054
57	Dimethylnaphthalene	38.725	0.009	1.276
58	Dimethylnaphthalene	38.921	0.028	0.710

This method requires no valve or precolumn. It only requires a normal GC equipped with split/splitless inlet, FID, and a single, polar HP-INNOWax capillary column. Quantitative results are improved using n-tridecane, instead of n-undecane, as the internal standard.

Conclusion

The aromatic hydrocarbons in finished gasoline, gasoline blending feedstock, and straight-run naphtha were quantitatively analyzed using an Agilent 6820 GC equipped with FID and a single polar capillary column. An internal standardization method, using n-tridecane as the internal standard, yielded quantitative concentration data for 57 aromatic target compounds, with a relative standard deviation of less than 1% for almost all compounds.

References

1. People's Republic of China National Standards GB 17930-1999.
2. ASTM D5769 ; the standard test method for determination of benzene, toluene, and total aromatics in finished gasoline by gas chromatography/mass spectrometry.
3. ASTM D5580 ; standard test method for determination of benzene, toluene, ethylbenzene, p/m-xylene, o-xylene, C9 and heavier aromatics and total aromatics in finished gasoline by GC.
4. ASTM D3606 ; standard test method for determination of benzene and toluene in finished motor and aviation gasoline by GC.
5. JIS K 2536-1996 ; Liquid petroleum products - testing method of components.

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